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Study of superconducting and electromagnetic properties of un-doped and organic compound doped MgB₂ conductors

Shahriar Al-Hossain
University of Wollongong

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**STUDY OF SUPERCONDUCTING AND ELECTROMAGNETIC
PROPERTIES OF UN-DOPED AND ORGANIC COMPOUND
DOPED MgB₂ CONDUCTORS**

A thesis submitted in fulfillment of the requirements for the award of the degree

DOCTOR OF PHILOSOPHY

From the

UNIVERSITY OF WOLLONGONG

By

MD. SHAHRIAR AL HOSSAIN, B.Sc.

Institute for Superconducting & Electronic Materials

Faculty of Engineering

2008

DECLARATION

This is to certify that the work presented in this thesis was carried out by the candidate in the laboratories of the Institute for Superconducting and Electronic Materials (ISEM), at the University of Wollongong, NSW, Australia, and has not been submitted for a degree to any other institution for higher education.

Md Shahriar Al Hossain

2008

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ABSTRACT

In this thesis I emphasized on the organic compound doping (specially carbohydrate group, malic acid, $C_4H_6O_5$) and heat treatment effects on the superconducting properties of MgB_2 . I also focused on the basic and fundamental properties of un-doped MgB_2 wires in different temperatures for comparison purpose. And finally I have proposed another new dopant which avoids some problems using carbohydrate in some aspects.

Firstly, I have studied the effects of sintering temperature on the phase transformation, lattice parameters, full width at half-maximum (FWHM), strain, critical temperature (T_c), critical current density (J_c) and resistivity (ρ) in MgB_2/Fe wires. All samples were fabricated by the *in situ* powder-in-tube method (PIT) and sintered within a temperature range of 650–900 °C. I have showed that why I have taken such sintering temperature range by analyzing with differential thermal analysis (DTA). The increased FWHM and decreased T_c at low sintering temperature region suggested the smaller grain size and poor crystallinity. Strain values also higher at low sintering region. That's why it was observed that wires sintered at low temperature, 650 °C, resulted in higher J_c up to 12 T. The best transport J_c value reached 4200 A cm⁻² at 4.2 K and 10 T. This is related to the grain boundary pinning due to small grain size and poor crystallinity due to strain defects. On the other hand, wires sintered at 900 °C had a lower J_c in combination with better crystallinity due to higher T_c .

The effect of carbohydrate doping on lattice parameters, microstructure, T_c , J_c , H_{irr} , and H_{c2} of MgB_2 has been studied. In this work I used malic acid, $C_4H_6O_5$ as an example of

carbohydrates as an additive to MgB_2 . We have described the advantages of carbohydrate doping include homogeneous mixing of precursor powders, avoidance of expansive nanoadditives, production of highly reactive C, and significant enhancement in J_c , H_{irr} , and H_{c2} of MgB_2 , compared to un-doped samples. The defects due to the C substitution into boron site lead to the enhancement of H_{irr} and H_{c2} . The decrease of a -axis lattice parameter and reduction of T_c indicates poor crystallinity due to C substitution. The microstructure was shown both for un-doped and doped samples which were well consistent with FWHM. The J_c for $\text{MgB}_2 + 30 \text{ wt\% C}_4\text{H}_6\text{O}_5$ sample was increased by a factor of 21 at 5 K and 8 T without degradation of self-field J_c due to C substitution into B sites.

During the evaporation process of the $\text{C}_4\text{H}_6\text{O}_5$ with B and solvent, freshly and highly reactive C is produced and C substitution for B can take place at the temperature same as the formation temperature of MgB_2 . By using this chemical route I again evaluated the doping effects of $\text{C}_4\text{H}_6\text{O}_5$, from 0 to 30 wt% of the total MgB_2 , on the lattice parameters, lattice strain, amount of carbon (C) substitution, microstructures, weight fraction of MgO, critical temperature (T_c), critical current density (J_c), and irreversibility field (H_{irr}) of a MgB_2 superconductor. The calculated lattice parameters show a large decrease in the a -axis lattice parameter for $\text{MgB}_2 + \text{C}_4\text{H}_6\text{O}_5$ samples from 3.0861(6) to 3.0736(1) Å, with even a 10 wt% addition. This is an indication of C substitution into boron sites, with the C coming from $\text{C}_4\text{H}_6\text{O}_5$, resulting in enhancement of J_c and H_{irr} . Specifically, the H_{irr} of the $\text{MgB}_2 + \text{C}_4\text{H}_6\text{O}_5$ samples prepared by the chemical solution route reached around 7 T at 20 K, with a T_c reduction of only 1.5 K. In addition, the self-field J_c of the $\text{MgB}_2 +$

$C_4H_6O_5$ samples was only slightly reduced at an additive level as high as 30 wt%. The interesting thing I found here is maximum C-substitution and the maximum enhancement of all the superconducting parameters up to 10 wt% addition, after that the improvement rate is saturated. From these data I can claim 10 wt% addition is enough for maximum C-substitution and enhancement of superconducting properties. However, residual oxygen after evaporation processing contributed to a large amount of MgO in our $MgB_2 + 30$ wt% $C_4H_6O_5$ samples. These problems can be further controlled by the amount of $C_4H_6O_5$ additive or different evaporation temperatures.

After the successful doping effects of $C_4H_6O_5$ into MgB_2 , then I investigated the behavior of $C_4H_6O_5$ as a dopant with different sintering temperatures. All the samples were prepared by the chemical solution route. I report the carbon (C) substitution effects of $MgB_2 + 10$ wt% $C_4H_6O_5$ on the lattice parameters, critical temperature (T_c), upper critical field (H_{c2}), and irreversibility field (H_{irr}) as a function of sintering temperature in the range from 600 to 900 °C. The additive $C_4H_6O_5$ as the C source resulted in a small depression in T_c , but significantly increased the C substitution level, and hence improved the H_{c2} and H_{irr} performance at a low sintering temperature of 600 °C in conjunction with a short sintering period of 4 h. In addition, the low-temperature sintering process resulted in small grain size and higher impurity scattering compared to a pure MgB_2 superconductor which promotes the flux pinning significantly.

Very recently, I have chosen another solid hydrocarbon dopant named pyrene ($C_{16}H_{10}$) in to MgB_2 . There are few reasons behind this decision. Firstly we know all the carbohydrates consist of carbon (C), hydrogen (H), and oxygen (O). During the

evaporation process of $C_4H_6O_5$, I noticed that the MgO amount is gradually increased with increasing doping level. So our group suggests such special solid hydrocarbon without O content which may reduce the MgO content within the matrix. In this work, we report on significantly enhanced J_c in MgB_2 superconductor that was easily obtained by doping with a hydrocarbon, highly active $C_{16}H_{10}$, and using a sintering temperature as low as 600 °C. The processing advantages of the $C_{16}H_{10}$ additive include production of a highly active carbon C source, an increased level of disorder, and the introduction of small grain size, resulting in enhancement of J_c .